GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION
SPONSORED PROJECT INITIATION

no action OK
Date: June 24, 1977

Project Title: Pyrolysis of Wood Samples

Project No: A-2015

Project Director: Dr. J. A. Knight

Sponsor: International Paper Company

Agreement Period: From 6/20/77 Until 8/1/77

Type Agreement: Std. Ind. Research

Amount: $5,500 (Includes $500 for data rights)

Reports Required: As requested.

Sponsor Contact Person (s):

Technical Matters

Contractual Matters (thru OCA)

Dr. J. Allen Cove, Mgr.
Wood Products Research
Corporate Research & Development Division
International Paper Company
P.O. Box 797
Tuxedo Park, New York 10987

Defense Priority Rating:

Assigned to: Technology & Development Laboratory (School/Laboratory)

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Reports Coordinator (OCA)	

CA-3 (3/76)

Ho action

GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION

SPONSORED PROJECT TERMINATION

Date: September 27, 1977

Project Title: Pyrolysis of Wood Samples

Project No: A-2015

Project Director: Dr. J. A. Knight

Sponsor: International Paper Company

Effective Termination Date: 9/13/77

Clearance of Accounting Charges: 9/30/77

Grant/Contract Closeout Actions Remaining:

- X Final Invoice ~~and Closing Documents~~
- _ Final Fiscal Report
- _ Final Report of Inventions
- _ Govt. Property Inventory & Related Certificate
- _ Classified Material Certificate
- _ Other ______

Assigned to: ~~Technology & Development Laboratory~~ (School/Laboratory)

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CA-4 (3/76)

Project A-2015

International Paper Company

PYROLYSIS OF WOOD SAMPLES

Report No. 1

Samples No. 1, 2, and 3

L. W. Elston and J. A. Knight

1. SUMMARY

Three samples of wood material have been pyrolyzed in the six-inch tube furnace apparatus. The resulting chars, oils, light oils and aqueous condensates are being forwarded under separate cover.

2. EXPERIMENTAL PROCEDURES

The tube furnace and its operation are described in Appendix A (attached). For these experiments the maximum temperature was 800° C., and the normal thermal gradient procedure was employed.

The off-gas collection train and its operation are described in Appendix B (attached). In these experiments the cold condensers were chilled by alcohol circulating through a bath of dry ice in ethylene glycol (approximately -40° C). As no gas composition data were required, the non-condensible gases were passed from the dry test meter to an exhaust fan rather than into gas collection bags.

3. ANALYTICAL PROCEDURES

3.1 Liquid Product Recovery

At the end of the run the off-gas collection train was disassembled, and the components were weighed. The aqueous phase components in the ice traps were separated from the heavier oil phases by decantation, and the traps containing the oil phase were reweighed. The weight of the aqueous phase component was calculated from the differences in these weighings. The weight of the organic phase was taken as the difference between the weights of the flasks containing the oil phases and their original (clean) weights. The aqueous fractions from the three traps were combined, mixed well, and stored under refrigeration in a tightly capped polyethylene bottle. The oil phases were poured and scraped from the three traps, combined, and stored under refrigeration in a tightly closed polyethylene bottle. This system of weights by difference allowed the reported yields to include the small amounts of oil phase which could not be scraped from the walls of the traps.

The demister condensate weight was calculated from the weight increase of the two glass wool demisters during the run.

The weight increase of the calcium sulfate dryer was determined.

The contents of the cold (dry ice) traps were poured into a tared polyethylene bottle, which was quickly capped and reweighed. This "light oil" fraction was stored in a freezer.

3.2 Moisture in Feeds

Moisture in feeds was determined by heating 100 gram samples overnight in tared shallow pans at 105° C in a forced air oven. Experience has shown that using large, samples of feed as received and well mixed offers the advantage of a representative sample and avoids the moisture loss in the grinding that would be required for a small representative sample.

3.3 Moisture in Liquid Condensates

The moisture content of the oil and aqueous phases is determined by azeotropic distillation with toluene for two hours.

4. RESULTS AND DISCUSSION

The results of the direct determinations described above are summarized in Table 1.

TABLE 1. Results of Direct Determinations

(All Weights in Grams)

Determination	Sample 1	Sample 2	Sample 3
Weight of Feed	3121	3764	3088
Percent Moisture in Feed	13.73	8.15	15.68
Char Weight	790	1040	741
Weight of Ice Trap Aqueous Phase	1165.9	1404.6	1283.0
Percent Moisture in Aqueous Phase	87.2	85.0	91.0
Weight of Ice Trap Organic Phase	192.1	246.3	132.9
Percent Moisture in Organic Phase	11.6	16.0	23.1
Weight of Demister Condensate	71.3	55.8	73.6
Weight of Dryer Condensate	27.7	17.9	17.9
Weight of Cold Trap (Light Oil) Condensate	41.7	59.4	46.2
Evolved Gas Volume (liters)	829	896	736

The data shown in Table 1 were used to calculate the materials balance data. It is hoped that presenting the details of the assumptions and calculations made in obtaining these data will be helpful in their evaluation. The values for Sample No. 1 will be used to illustrate the steps which were used for each of the samples.

$$\text{Weight of Dry Feed} = \text{Feed Weight} \times \left(1 - \frac{\text{percent moisture}}{100}\right)$$

$$\text{Weight of Dry Feed} = 3121 \times \left(1 - \frac{13.73}{100}\right) = 2692.5 \text{ grams}$$

Weight of Water Input = Weight of Feed-Weight of Dry Feed
Weight of Input Water = 3121 - 2692.5 = 428.5 grams

$$\text{"Dry" Weight of Ice Trap Oil Phase} = \text{Weight of Oil Phase} \times \left(1 - \frac{\text{percent moisture}}{100}\right)$$

$$\text{"Dry" Weight of Ice Trap Oil} = 192.1 \times \left(1 - \frac{11.6}{100}\right) = 169.8 \text{ grams}$$

Weight of moisture in Ice Trap Oil = 192.1 - 169.8 = 22.3 grams

$$\text{Weight of Water in Aqueous Phase} = \frac{\text{percent moisture}}{100} \times \text{Weight of Aqueous Phase}$$

$$= 1165.9 \times \frac{87.2}{100} = 1016.7 \text{ grams}$$

Weight of Dissolved/Dispersed Oil = 1165.9 - 1016.7 = 149.2 grams

Total "Dry" Oil in Ice Traps = Dry Organic Phase + Dissolved/Dispersed Oil
Total "Dry" Oil in Ice Traps = 169.8 + 149.2 = 319 grams

Total Water in Ice Traps = Organic Phase Water + Aqueous Phase Water
Total Water in Ice Traps = 1016.7 + 22.3 = 1039 grams

It was assumed that the ratio of oil to water in the demister condensate is approximately equal to that in the ice trap condensates.

$$\text{Weight of "Dry" Oil in Demisters} = \frac{\text{Weight of Trap Dry Oil}}{\text{Weight of Trap Condensates}} \times \text{Weight of Demister Condensates}$$

$$= \frac{319}{319 + 1039} \times 71.3 = 16.7 \text{ grams}$$

Weight of Water in Demisters = 71.3 - 16.7 = 54.6 grams

The weight of the condensate in the calcium sulfate dryer was assumed to be all water.

Total Oil Collected = Trap Oils + Demister Oil:
= 319 + 16.7 = 355.7 grams

Total Water Collected = Trap Water + Demister Water + Dryer Water
= 1039 + 54.6 + 27.7 = 1121.3 grams

Weight of Water Yield = Total Water Collected - Input Water
= 1121.9 - 428.5 = 693.4

The collected results of these calculations are summarized in Table 2.

TABLE 2. Calculated Input and Yield Weights (grams)

	Sample 1	Sample 2	Sample 3
Feed Input (Water Free)	2692.5	3457.2	2603.8
Water Input (In Feed)	428.5	306.8	484.2
Char Yield	790	1040	741
Organic Phase Oil Yield (Water Free)	169.8	206.9	132.9
Dissolved/Dispersed Oil Yield (Water Free)	149.2	210.7	115.5
Demister Oil (Water Free)	16.7	14.1	12.6
Total Oil Yield (Sum of Above Oils)	335.7	431.7	261.0
Water Collected	1121.3	1292.9	1286.3
Water Yield	692.8	986.1	802.1
Light Oil (Cold Trap) Yield	41.7	59.4	46.2
Gas Yield (By Difference)	832.3	940.0	753.5

The percent yield data are shown in Table 3. These yields are based on the calculated "water free" weights of all the feeds and products, except the water yield value.

TABLE 3. Percent Yields Dry Feed Basis

	Sample 1	Sample 2	Sample 3
Char	29.3	30.1	28.5
Oil	12.5	12.5	10.0
Light Oil	1.5	1.7	1.8
Water	25.7	28.5	30.8
Noncondensible Gas	30.9	27.2	28.9

2. OPERATION

After thorough cleaning, the upstream end of the tube, including spacer and thermocouples, is assembled; and the weighed charge of feed is loaded through the open downstream end of the tube and packed. The downstream spacer, thermocouples and end cap are then added; and the assembled pyrolysis tube is placed in the cold furnace and connected to the condensate and gas collection train. The entire assembly is then tested for leaks. If desired, the assembly is purged with an inert gas (usually prepurified nitrogen) admitted through a fitting in the upstream end plate to sweep the air from the system.

In a normal pyrolysis the upstream furnace zone (thermocouple No. 1) and the slower center zone (thermocouple No. 2) are set at the desired pyrolysis temperature, and the furnace is turned on. The downstream zone (thermocouple No. 3) is left at its minimum (200° C) setting until the temperature at the center of the charge (thermocouple No. 6) reaches its maximum value. As the center of the charge reaches its maximum temperature, power is applied to the downstream furnace zone to raise it to the desired final temperature and complete the pyrolysis.

This procedure creates a thermal gradient. The liquids and gases flow from their origin in the hotter part of the furnace through a relatively cooler part of the tube to the exit so that thermal cracking is minimized during most of the run.

The pyrolysis procedure is usually continued until the rate of gas evolution approaches zero. At the end of the run the ball valve on the exit tube is closed to exclude air during cooling, and the furnace power is turned off. The solid product (char) is usually removed from the cooled tube on the day following the run.

Details of the off-gas collection system are given in a separate appendix.

APPENDIX A

SIX-INCH TUBE FURNACE APPARATUS

1. APPARATUS

The six-inch pyrolysis unit consists of a five-foot length of Schedule 80 six-inch stainless steel pipe heated by a three-zone Lindberg tube furnace. The ends are closed by means of heavy aluminum plates tightly compressed against silicone rubber gaskets. Stainless steel spacers are provided to confine the feed material to the uniformly heated center zone of the apparatus. The temperatures of the three separately controlled furnace zones and of selected locations within the tube are measured by chromel-alumel thermocouples and recorded. A schematic diagram of the tube furnace arrangement is shown in Figure 1, with location of thermocouples numbered 1 through 10.

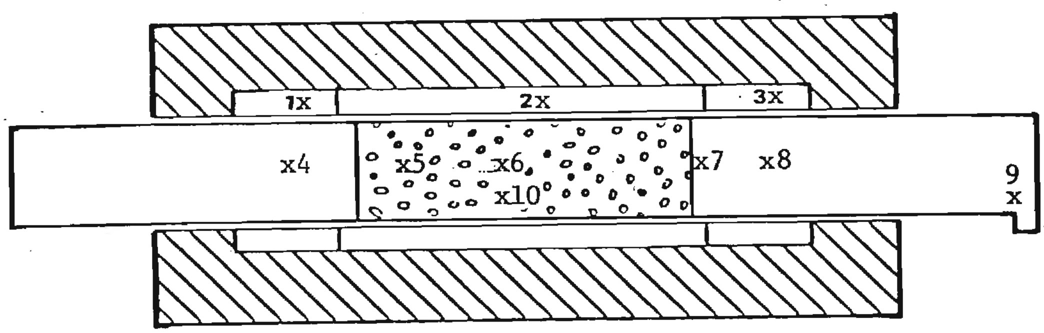

Figure 1. Schematic Diagram of Tube Furnace

The upstream end of the apparatus (left hand end in the diagram) is raised slightly to promote gravity flow of liquid products toward the one-inch ID stainless steel exit tube located at the bottom of the downstream end of the pyrolysis tube. The spacer on the downstream end of the charge is slotted at the bottom to permit liquid flow. The exit tube ends in a one-inch stainless steel Whitey ball valve. This valve, which is used to exclude air from the pyrolized charge during cooling, is fitted to accept the upstream end of the condensate collection train.

APPENDIX B

TUBE OFF-GAS COLLECTION TRAIN

1. APPARATUS

A schematic diagram of the train is shown in Figure 2.

Liquids and gases emerge from the pyrolysis tube through a stainless steel ball valve (1) into a series of water cooled condensers (2) and ice cooled traps (3). The first condenser is a jacketed stainless steel tube, which minimizes the risk of breakage that might occur in a heated metal-to-glass joint. The first trap is a resin kettle rather than a flask so that viscous condensates may easily be recovered. The gas stream then passes through a glass wool demister (4) and a calcium sulfate ("Drierite) column (5) into a series of cold condensers (6) and cold "light oil" traps (7). The condensers are chilled by ethanol circulating through a heat exchanger coil immersed in dry ice and ethylene glycol for most experiments or in dry ice and acetone when a large quantity of hydrogen sulfide is anticipated. The traps are immersed in a bath of dry ice and acetone. From the cold traps, the gases pass through a magnesium perchlorate drier (8) and a calibrated dry test meter (9) into a series of 96 liter gas collection bags. The quantity of magnesium perchlorate, which is necessary to prevent subsequent fouling of gas chromatographic columns, is held to a minimum to reduce possible explosion hazards.

2. OPERATION

After assembly and thorough leak testing, the train is connected to the pyrolysis tube and the refrigerants are added. The ball valve is opened at the start of the run and closed when the run is completed. During the run 90 liter quantities of non-condensible gas are collected successively in a series of 96 liter gas collection bags.

After each bag is filled, it is kneaded to mix its contents and then emptied by aspiration through a gas collection tube. When the bag is approximately half-emptied the gas collection tube is closed and labelled for laboratory analysis. If sulfur gases are of interest a measured portion of the of the gas is drawn through a special sulfur gas absorption train. The remainder of the gas from each bag is then pumped to an exhaust fan.

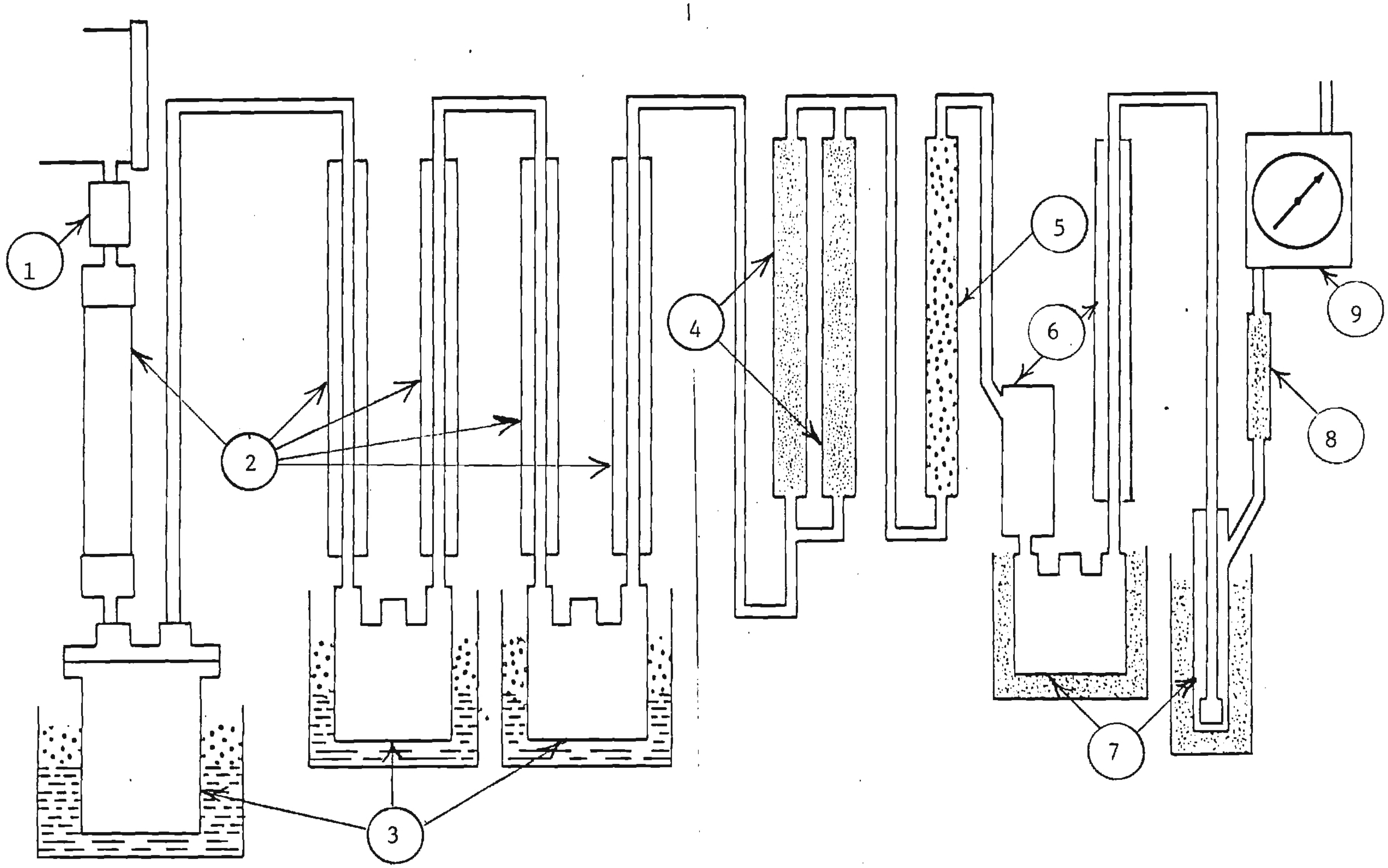

Figure 2. Schematic Diagram of Condensation Train

On completion of the run the ball valve is closed and the weights of the condensates are determined. The condensates are then transferred to tightly closed containers and transported to the wet chemistry laboratory for analysis. The heavy organic and aqueous condensates are stored in a refrigerator. The light oils (from the dry ice traps) are stored in a freezer.

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

August 24, 1977

Dr. J. Allan Love
Manager
Wood Products Research
Corporate Research & Dev. Div.
International Paper Company
P.O. Box 797
Tuxedo Park, N.Y. 10987

#2

Subject: Report, Project A-2015, "Pyrolysis of Wood Samples," Samples 4, 5 and 6

Dear Allan:

The report for samples 4, 5 and 6 are enclosed. We enjoyed having Dr. Fu visit with us and hope that it was of value to him.

If we can supply you with any additional information about the report, please contact Pete Elston or me.

Sincerely,

James A. Knight, HEAD
RESOURCE UTILIZATION BRANCH

JAK:bc

Enclosures (3)

Project A-2015

INTERNATIONAL PAPER COMPANY
PYROLYSIS OF WOOD SAMPLES

Report No. 2
Samples No. 4, 5, and 6

L. W. Elston and J. A. Knight

1. SUMMARY

Samples of 4, 5, and 6 of the wood materials have been pyrolized in the six-inch tube furnace. The resulting chars from Samples No. 1, 2, 3, 4, 5 and 6 have been forwarded to your laboratories by UPS. The aqueous and oil condensates were delivered to Dr. Yun-Lung Fu on his visit to our laboratory on August 18, 1977.

2. EXPERIMENTAL AND ANALYTICAL PROCEDURES

The experimental and analytical procedures described in Progress Report No. 1 have been followed rigorously.

3. RESULTS AND DISCUSSION

The results for samples 4, 5, and 6 are summarized in Tables No. 1, 2, and 3 corresponding to the similarly labelled tables in Progress Report No. 1. The assumptions and methods of calculation are the same as those previously described in Progress Report No. 1.

Table 1. Results of Direct Determinations
(all weights in grams)

Determination	Sample 4	Sample 5	Sample 6
Weight of feed	2256	3879	2628
Percent moisture in feed	9.0	10.45	9.92
Char weight	654	890	658
Weight of ice trap aqueous phase	928.2	1385.8	1025.3

Table 1. Results of Direct Determinations (cont'd.)

(all weights in grams)

Determination	Sample 4	Sample 5	Sample 6
Percent moisture in aqueous phase	85.4	86.9	84.1
Weight of ice trap organic phase	90.8	294.2	131.1
Percent moisture in organic phase	17.2	19.5	15.7
Weight of demister condensate	51.0	80.9	74.4
Weight of dryer condensate	14.5	23.5	16.8
Weight of cold trap (light oil) Condensate	29.7	60.0	40.7
Evolved gas volume (liters)	553	895	617

Table 2. Calculated Input and Yield Weights

(grams)

Determination	Sample 4	Sample 5	Sample 6
Feed input (water free)	2053.0	3473.6	2367.3
Water input (in feed)	203.0	405.4	260.7
Char yield	654	890	658
Organic phase oil yield (water free)	75.2	236.8	110.5
Dissolved/dispersed oil yield (water free)	135.5	181.5	163.0
Demister oil (water free)	10.5	20.1	17.6
Total oil yield (sum of above oils)	221.2	438.4	291.1
Water Collected	863.3	1346.0	917.3
Water yield	660.3	940.6	656.6
Light oil (cold trap) yield	29.7	60.0	40.7
Gas yield (by difference)	487.8	1144.6	720.9

Table 3. Percent Yields (Dry Feed Basis)

Determination	Sample 4	Sample 5	Sample 6
Char	31.9	25.6	27.8
Oil	10.8	12.6	12.3
Light oil	1.4	1.7	1.7
Water	32.2	27.1	27.7
Noncondensible gas	23.7	33.0	30.5

A-2015

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

Atlanta, Georgia

Final Report

June 20, 1977 - September 18, 1977

Project A-2015

PYROLYSIS OF WOOD SAMPLES

by

L. W. Elston and J. A. Knight

Performed for

International Paper Company
Wood Products Research
Corporate Research & Dev. Div.
Tuexdo Park, New York 10987

FINAL REPORT

L. W. Elston and J. A. Knight

Summary of Work and Reports

The results with samples 1, 2 and 3 were given in Report No. 1, which were forwarded to International Paper August 12, 1977.

The results with samples 4, 5 and 6 were given in Report No. 2, which were forwarded to International Paper, August 24, 1977.

Samples 7, 8, 9 and 10 have been completed and the results are given in Report No. 3, which is enclosed as part of this final report. Report No. 3 also contains the time-temperature printouts of the thermocouples in the tube furnace. In runs 8 and 9, malfunctions were experienced with the recorder, which are noted on the printouts. For run 10, the temperatures were manually recorded. Under separate cover, copies of the time-temperature printouts for samples 1 through 6 were sent to Dr. Yun-Lung Fu on August 25, 1977.

PROJECT A-2015

INTERNATIONAL PAPER COMPANY
PYROLYSIS OF WOOD SAMPLES

Report No. 3 and Final Report
Samples No. 7, 8, 9, and 10

L. W. Elston and J. A. Knight

1. SUMMARY

Portions of Samples No. 7, 8, 9, and 10 have been pyrolyzed in the six-inch tube furnace. The condensates, packed in dry ice, and chars have been forwarded to your laboratory by air express.

2. EXPERIMENTAL AND ANALYTICAL PROCEDURES

The experimental and analytical procedures described in Progress Report No. 1 have been followed rigorously.

3. RESULTS AND DISCUSSION

The results for Samples 7, 8, 9, and 10 are summarized in Tables No. 1, 2, and 3 corresponding to the similarly labelled tables in Progress Reports No. 1 and 2. The assumptions and methods of calculation are the same as those previously described in Progress Report No. 1.

Examination of the data in Table 3 indicates that the char and gas yields for Sample No. 10 are higher than those for the other samples, while the oil and water yields are somewhat less.

The recorded time/temperature data are incomplete, as the printing digital recorder failed. However, the temperatures of the thermocouples as shown by the digital readouts connected in parallel with the printing recorder indicated normal temperature rise patterns. The data printouts for Sample No. 7 are complete and coincide with the digital readouts. The malfunctions are noted on the printouts for Samples 8 and 9. The times and evolved gas data noted on these printouts are correct. Regretfully, the temperature rises were monitored on the digital readouts, but not fully recorded. For Sample No. 10, the data were read from the digital printouts and recorded manually during the entire run.

Table 1. Results of Direct Determinations

(All weights in grams)

Determination	Sample 7	Sample 8	Sample 9	Sample 10
Weight of Feed	3324	2530	3126	3434
Percent Moisture in Feed	10.83	18.17	9.98	10.02
Char Weight	879	555	832	1097
Weight of Ice Trap Aqueous Phase	1186.6	1061.6	1130.0	987.5
Percent Moisture in Aqueous Phase	88.6	93.61	88.66	95.40
Weight of Ice Trap Organic Phase	261.9	135.1	200.3	152.2
Percent Moisture in Organic Phase	27.77	11.44	15.31	16.00
Weight of Demister Condensate	79.2	70.6	72.5	71.1
Weight of Dryer Condensate	16.4	18.3	18.5	23.0
Weight of Cold Trap (Light Oil) Condensate	48.7	38.0	55.9	48.0
Evolved Gas Volume (Liters)	857	680	829	1007

Table 2. Calculated Input and Yield Weights (grams)

Determination	Sample 7	Sample 8	Sample 9	Sample 10
Feed Input (Water Free)	2964.0	2070.3	2814.0	3089.9
Water Input (In Feed)	360.0	459.7	312.0	344.1
Char Yield	879	555	829	1007
Organic Phase Oil Yield (Water Free)	189.2	119.6	169.7	127.8
Dissolved/Dispersed Oil (Water Free)	135.3	67.8	128.1	45.4
Demister Oil (Water Free)	17.7	11.1	16.2	12.8
Total Oil Yield (Sum of above oils)	342.2	198.5	314.0	186.0
Water Collected	1201.9	1087.1	1107.3	1060.6
Water Yield	841.9	627.4	795.3	716.5
Light Oil (Cold Trap) Yield	48.7	38.1	55.9	48.0
Gas Yield (by difference)	852.2	651.3	819.8	1132.4

Table 3. Percent Yields (Dry Feed Basis)

Determination	Sample 7	Sample 8	Sample 9	Sample 10
Char	29.7	26.8	29.5	32.6
Oil	11.5	9.6	11.2	6.0
Light Oil	1.6	1.8	2.0	1.6
Water	28.4	30.3	28.3	23.2
Noncondensible Gases	28.8	31.5	29.1	36.7

Sample 7, Page 1

0:10

11	0074	F
10	0105	F
9	0084	F
8	0098	F
7	0082	F
6	0111	F
5	0078	F
4	0082	F
3	0384	F
2	0341	F
1	0636	F
0	0076	F

0:05

11	0073	F
10	0085	F
9	0079	F
8	0083	F
7	0081	F
6	0086	F
5	0078	F
4	0080	F
3	0393	F
2	0224	F
1	0393	F
0	0075	F

8-18-77
Run #7
A2015
Time 00:00
Meter 0.0

11	0073	F
10	0079	F
9	0077	F
8	0078	F
7	0078	F
6	0077	F
5	0074	F
4	0078	F
3	0077	F
2	0077	F
1	0074	F
0	0073	F

0:25

11	0076	F
10	0228	F
9	0110	F
8	0143	F
7	0087	F
6	0263	F
5	0089	F
4	0122	F
3	0503	F
2	0631	F
1	1037	F
0	0078	F

0:20

11	0075	F
10	0177	F
9	0100	F
8	0127	F
7	0085	F
6	0201	F
5	0083	F
4	0097	F
3	0450	F
2	0549	F
1	0928	F
0	0078	F

0:15

11	0074	F
10	0136	F
9	0092	F
8	0112	F
7	0083	F
6	0149	F
5	0080	F
4	0086	F
3	0405	F
2	0455	F
1	0800	F
0	0077	F

Sample 7 Page 2

TIME 00:42
M= 16 liters

11	0077	F
10	0437	F
9	0144	F
8	0201	F
7	0199	F
6	0489	F
5	0207	F
4	0213	F
3	0627	F
2	0834	F
1	1264	F
0	0078	F

0:36

11	0076	F
10	0355	F
× 9		F
8	0177	F
7	0106	F
6	0411	F
5	0188	F
4	0208	F
3	0591	F
2	0775	F
1	1203	F
0	0078	F

0:31

11	0076	F
10	0287	F
9	0119	F
8	0159	F
7	0094	F
6	0332	F
5	0110	F
4	0170	F
3	0548	F
2	0707	F
1	1131	F
0	0078	F

0:57

11	0077	F
10	0717	F
9	0251	F
8	0330	F
7	0207	F
6	0757	F
5	0250	F
4	0519	F
3	0718	F
2	0990	F
1	1435	F
0	0079	F

0:52

11	0077	F
10	0632	F
9	0217	F
8	0290	F
7	0205	F
6	0666	F
5	0226	F
4	0364	F
3	0691	F
2	0940	F
1	1384	F
0	0078	F

0:47

11	0077	F
10	0535	F
× 9		F
8	0245	F
7	0206	F
6	0574	F
5	0215	F
4	0235	F
3	0660	F
2	0889	F
1	1327	F
0	0079	F

Sample 7, Page 3

01:13

176

11	0077	F
10	0897	F
9	0327	F
8	0471	F
7	0213	F
6	0959	F
5	0406	F
4	0634	F
3	0795	F
2	1116	F
1	1476	F
0	0078	F

01:06

11	0077	F
10	0847	F
9	0304	F
8	0429	F
7	0210	F
6	0904	F
5	0338	F
4	0617	F
3	0769	F
2	1076	F
1	1477	F
0	0078	F

01:05

11	0077	F
10	0787	F
9	0278	F
8	0378	F
7	0209	F
6	0836	F
5	0288	F
4	0598	F
3	0744	F
2	1036	F
1	1476	F
0	0079	F

1:29

11	0078	F
10	1044	F
9	0405	F
8	0583	F
7	0224	F
6	1109	F
5	0777	F
4	0778	F
3	0873	F
2	1230	F
1	1475	F
0	0079	F

~~0:23~~

1:23

11	0077	F
10	0994	F
9	0381	F
8	0566	F
7	0220	F
6	1061	F
5	0621	F
4	0714	F
3	0847	F
2	1192	F
1	1476	F
0	0078	F

1:18

11	0077	F
10	0946	F
9	0348	F
8	0524	F
7	0215	F
6	1011	F
5	0500	F
4	0665	F
3	0823	F
2	1155	F
1	1476	F
0	0079	F

Sample 7, Page 4

01:44
509

11	0078	F
10	1191	F
9	0473	F
8	0645	F
7	0260	F
6	1241	F
5	1241	F
4	0984	F
3	0950	F
2	1341	F
1	1475	F
0	0078	F

1:39

11	0078	F
10	1143	F
9	0455	F
8	0629	F
7	0242	F
6	1198	F
5	1117	F
4	0912	F
3	0925	F
2	1304	F
1	1476	F
0	0079	F

01:34
379

11	0077	F
10	1095	F
9	0430	F
8	0608	F
7	0231	F
6	1154	F
5	0971	F
4	0844	F
3	0899	F
2	1268	F
1	1475	F
0	0079	F

01:59
692

11	0078	F
10	1336	F
9	0501	F
8	0690	F
7	0592	F
6	1359	F
5	1446	F
4	1263	F
3	1022	F
2	1451	F
1	1475	F
0	0080	F

01:54
633

11	0078	F
10	1286	F
9	0494	F
8	0672	F
7	0397	F
6	1322	F
5	1425	F
4	1154	F
3	0999	F
2	1415	F
1	1476	F
0	0080	F

01:49
573

11	0078	F
10	1238	F
9	0482	F
8	0654	F
7	0303	F
6	1281	F
5	1364	F
4	1061	F
3	0975	F
2	1378	F
1	1475	F
0	0079	F

Sample 7, Page 5

02:15
793

11	0078	F
10	1421	F
9	0514	F
8	0705	F
7	1147	F
6	1424	F
5	1481	F
4	1467	F
3	1077	F
2	1475	F
1	1474	F
0	0080	F

2:30

11	0078	F
10	1422	F
9	0627	F
8	0957	F
7	1229	F
6	1418	F
5	1469	F
4	1443	F
3	1363	F
2	1470	F
1	1474	F
0	0080	F

02:10
775

11	0078	F
10	1418	F
9	0508	F
8	0695	F
7	1044	F
6	1426	F
5	1482	F
4	1466	F
3	1048	F
2	1482	F
1	1474	F
0	0080	F

02:25
811

11	0079	F
10	1419	F
9	0571	F
8	0853	F
7	1209	F
6	1418	F
5	1469	F
4	1447	F
3	1278	F
2	1469	F
1	1474	F
0	0080	F

2:05

11	0078	F
10	1386	F
9	0505	F
8	0695	F
7	0847	F
6	1401	F
5	1467	F
4	1385	F
3	1043	F
2	1483	F
1	1474	F
0	0080	F

2:20

11	0079	F
10	1419	F
9	0531	F
8	0760	F
7	1193	F
6	1420	F
5	1474	F
4	1454	F
3	1189	F
2	1470	F
1	1474	F
0	0081	F

Sample 7, Page 6

11	0079	F
10	1431	F
9	0822	F
8	1184	F
7	1333	F
6	1426	F
5	1463	F
4	1446	F
3	1471	F
2	1470	F
1	1474	F
0	0080	F

2:46

11	0079	F
10	1427	F
9	0765	F
8	1140	F
7	1297	F
6	1424	F
5	1464	F
4	1445	F
3	1469	F
2	1469	F
1	1474	F
0	0081	F

02:41
829

11	0079	F
10	1425	F
9	0693	F
8	1057	F
7	1260	F
6	1421	F
5	1467	F
4	1444	F
3	1437	F
2	1470	F
1	1474	F
0	0081	F

02:35
823

11	0078	F
10	1435	F
9	0913	F
8	1238	F
7	1385	F
6	1432	F
5	1453	F
4	1448	F
3	1471	F
2	1469	F
1	1475	F
0	0080	F

3:02

11	0078	F
10	1434	F
9	0892	F
8	1227	F
7	1374	F
6	1431	F
5	1456	F
4	1447	F
3	1472	F
2	1469	F
1	1474	F
0	0080	F

02:57
843.5

11	0078	F
10	1432	F
9	0864	F
8	1210	F
7	1358	F
6	1429	F
5	1459	F
4	1447	F
3	1472	F
2	1469	F
1	1474	F
0	0080	F

2:52

Sample 7 Page 7

03:17
853.5

11	0079	F
10	1437	F
9	0948	F
8	1256	F
7	1397	F
6	1434	F
5	1445	F
4	1447	F
3	1471	F
2	1469	F
1	1474	F
0	0081	F

3:12

11	0078	F
10	1437	F
9	0939	F
8	1252	F
7	1395	F
6	1433	F
5	1448	F
4	1447	F
3	1470	F
2	1469	F
1	1474	F
0	0080	F

03:07
849.5

11	0079	F
10	1436	F
9	0928	F
8	1247	F
7	1390	F
6	1433	F
5	1451	F
4	1447	F
3	1471	F
2	1469	F
1	1474	F
0	0081	F

THE END

03:27
856.5

11	0079	F
10	1438	F
9	0960	F
8	1262	F
7	1402	F
6	1435	F
5	1441	F
4	1446	F
3	1470	F
2	1469	F
1	1474	F
0	0081	F

03:22
855

11	0080	F
10	1437	F
9	0954	F
8	1260	F
7	1400	F
6	1434	F
5	1444	F
4	1446	F
3	1469	F
2	1469	F
1	1474	F
0	0080	F

Sample 8, Page 1

0:10

11	0081	F
10	0089	F
9	0086	F
8	0099	F
7	0083	F
6	0085	F
5	0078	F
4	0084	F
3	0387	F
2	0349	F
1	0641	F
0	0082	F

0:05

11	0081	F
10	0083	F
9	0082	F
8	0085	F
7	0083	F
6	0082	F
5	0078	F
4	0082	F
3	0396	F
2	0226	F
1	0400	F
0	0082	F

TIME=00:00
GASMETER-0.0

11	0080	F
10	0082	F
9	0080	F
8	0081	F
7	0083	F
6	0081	F
5	0078	F
4	0082	F
3	0082	F
2	0082	F
1	0081	F
0	0082	F

0:26

11	0081	F
10	0161	F
9	0111	F
8	0144	F
7	0088	F
6	0170	F
5	0096	F
4	0174	F
3	0508	F
2	0641	F
1	1043	F
0	0083	F

00:21
4 liter

11	0081	F
10	0133	F
9	0102	F
8	0129	F
7	0084	F
6	0129	F
5	0083	F
4	0128	F
3	0457	F
2	0559	F
1	0936	F
0	0082	F

0:15

11	0081	F
10	0106	F
9	0094	F
8	0114	F
7	0083	F
6	0098	F
5	0080	F
4	0095	F
3	0410	F
2	0465	F
1	0811	F
0	0082	F

Sample 8, Page 2

00:42
18

11	0082	F
10	0227	F
9	0154	F
8	0217	F
7	0208	F
6	0207	F
5	0207	F
4	0215	F
3	0635	F
2	0844	F
1	1280	F
0	0083	F

0:37

11	0081	F
10	0206	F
9	0133	F
8	0181	F
7	0113	F
6	0207	F
5	0196	F
4	0215	F
3	0598	F
2	0784	F
1	1209	F
0	0083	F

0:31

11	0082	F
10	0185	F
9	0122	F
8	0162	F
7	0094	F
6	0199	F
5	0128	F
4	0205	F
3	0556	F
2	0709	F
1	1137	F
0	0083	F

0:57

11	0080	F
10	0448	F
9	0241	F
8	0294	F
7	0203	F
6	0542	F
5	0248	F
4	0569	F
3	0730	F
2	1005	F
1	1451	F
0	0081	F

00:52
41

11	0082	F
10	0341	F
9	0216	F
8	0271	F
7	0204	F
6	0410	F
5	0231	F
4	0441	F
3	0702	F
2	0956	F
1	1396	F
0	0083	F

0:47

11	0081	F
10	0265	F
9	0203	F
8	0249	F
7	0205	F
6	0240	F
5	0216	F
4	0260	F
3	0670	F
2	0897	F
1	1342	F
0	0083	F

Sample 8, Page 3

01:13
155

11	0056	F
10	0602	F
9	0275	F
8	0370	F
7	0091	F
6	0672	F
5	0251	F
4	0634	F
3	0754	F
2	1028	F
1	1378	F
0	0056	F

01:48
497

11	0007	F
10	0574	F
9	0013	F
8	0147	F
7	0005	F
6	0597	F
5	0792	F
4	0640	F
3	0394	F
2	0818	F
1	0943	F
0	0000	F

107
RECORDER MALFUNCTION BEGINS

11	0034	F
10	0588	F
9	0211	F
8	0264	F
7	0119	F
6	0610	F
5	0220	F
4	0603	F
3	0621	F
2	1033	F
1	1403	F
0	0027	F

01:43
444

11	0000	F
10	0594	F
9	0032	F
8	0219	F
7	0002	F
6	0561	F
5	0777	F
4	0614	F
3	0431	F
2	0852	F
1	0932	F
0	0002	F

1:02

11	0081	F
10	0550	F
9	0266	F
8	0325	F
7	0200	F
6	0609	F
5	0277	F
4	0608	F
3	0757	F
2	1050	F
1	1473	F
0	0081	F

01:23
236

11	0001	F
10	0783	F
9	0256	F
8	0403	F
7	0065	F
6	0779	F
5	0627	F
4	0808	F
3	0723	F
2	1134	F
1	1390	F
0	0037	F

Sample 8, Page 4

~~02:20~~
646

11	0000	F
10	0756	F
9	0032	F
8	0147	F
7	0464	F
6	0798	F
5	0812	F
4	0809	F
3	0482	F
2	0844	F
1	0825	F
0	0002	F

02:15
643

11	0005	F
10	0728	F
9	0004	F
8	0045	F
7	0456	F
6	0678	F
5	0689	F
4	0747	F
3	0304	F
2	0697	F
1	0776	F
0	0008	F

02:51
670

11	0000	F
10	0611	F
9	0061	F
8	0421	F
7	0501	F
6	0589	F
5	0619	F
4	0628	F
3	0603	F
2	0564	F
1	0689	F
0	0000	F

01:59
585

11	0000	F
10	0730	F
9	0025	F
8	0111	F
7	0021	F
6	0711	F
5	0855	F
4	0787	F
3	0450	F
2	0896	F
1	0942	F
0	0001	F

02:30
654

11	0009	F
10	0829	F
9	0029	F
8	0288	F
7	0627	F
6	0772	F
5	0868	F
4	0783	F
3	0749	F
2	0860	F
1	0849	F
0	0011	F

Sample 8, Page 5

11	0000	F
10	0579	F
9	0120	F
8	0350	F
7	0497	F
6	0518	F
5	0538	F
4	0553	F
3	0511	F
2	0527	F
1	0583	F
0	0001	F

03:10
680

11	0003	F
10	0639	F
9	0004	F
8	0365	F
7	0485	F
6	0587	F
5	0552	F
4	0523	F
3	0636	F
2	0518	F
1	0634	F
0	0001	F

3:06

11	0000	F
10	0521	F
9	0083	F
8	0340	F
7	0500	F
6	0404	F
5	0531	F
4	0529	F
3	0532	F
2	0395	F
1	0627	F
0	0000	F

03:01
676

Sample 9, Page 1

0:10

11	0078	F
10	0088	F
9	0091	F
8	0105	F
7	0088	F
6	0089	F
5	0084	F
4	0103	F
3	0388	F
2	0352	F
1	0638	F
0	0082	F

0:05

11	0079	F
10	0087	F
9	0087	F
8	0091	F
7	0088	F
6	0087	F
5	0084	F
4	0089	F
3	0398	F
2	0227	F
1	0407	F
0	0082	F

TIME 00:00
METER 0

11	0006	F
10	0051	F
9	0011	F
8	0035	F
7	0016	F
6	0019	F
5	0005	F
4	0017	F
3	0046	F
2	0066	F
1	0061	F
0	0054	F

0:25

11	0079	F
10	0112	F
9	0116	F
8	0153	F
7	0095	F
6	0170	F
5	0096	F
4	0224	F
3	0508	F
2	0641	F
1	1042	F
0	0083	F

0:20

11	0080	F
10	0097	F
9	0108	F
8	0137	F
7	0090	F
6	0128	F
5	0088	F
4	0173	F
3	0458	F
2	0559	F
1	0945	F
0	0083	F

00:15
2 liters

11	0079	F
10	0090	F
9	0099	F
8	0122	F
7	0089	F
6	0098	F
5	0085	F
4	0132	F
3	0409	F
2	0466	F
1	0810	F
0	0082	F

8-24-77
A2015
#9

Sample 9, Page 2

0:41

11	0078	F
10	0201	F
9	0154	F
8	0221	F
7	0200	F
6	0215	F
5	0208	F
4	0447	F
3	0628	F
2	0843	F
1	1274	F
0	0082	F

0:36

11	0080	F
10	0160	F
9	0138	F
8	0190	F
7	0148	F
6	0205	F
5	0185	F
4	0358	F
3	0595	F
2	0784	F
1	1214	F
0	0083	F

00:31
9 liters

11	0080	F
10	0134	F
9	0127	F
8	0170	F
7	0110	F
6	0196	F
5	0118	F
4	0285	F
3	0553	F
2	0716	F
1	1133	F
0	0083	F

00:57
80

11	0008	F
10	0000	F
9	0014	F
8	0037	F
7	0001	F
6	0084	F
5	0035	F
4	0222	F
3	0166	F
2	0435	F
1	0906	F
0	0008	F

0:52

11	0008	F
10	0000	F
9	0014	F
8	0034	F
7	0001	F
6	0063	F
5	0032	F
4	0220	F
3	0277	F
2	0481	F
1	0975	F
0	0007	F

00:47
30 liters

11	0009	F
10	0027	F
9	0105	F
8	0138	F
7	0039	F
6	0187	F
5	0063	F
4	0398	F
3	0518	F
2	0694	F
1	1228	F
0	0046	F

Begin Recorder Malfunction

Sample 9, Page 3.

01:43
567

11	0000	F
10	-0018	F
9	0002	F
8	-0013	F
7	0001	F
6	0022	F
5	0001	F
4	0026	F
3	0006	F
2	0028	F
1	0015	F
0	-0000	F

2:35

1434
860
1206
1329
1437
1473
1454
1475
1476
1481

11	-0000	F
10	0031	F
9	0017	F
8	0018	F
7	0028	F
6	-0031	F
5	0032	F
4	0031	F
3	0032	F
2	0032	F
1	0033	F
0	0000	F

01:28
375

11	0000	F
10	0042	F
9	0007	F
8	0005	F
7	0005	F
6	0072	F
5	0002	F
4	0133	F
3	0072	F
2	0117	F
1	0491	F
0	0001	F

02:15
784

11	-0000	F
10	0030	F
9	0011	F
8	0016	F
7	0009	F
6	0031	F
5	0032	F
4	0031	F
3	-0025	F
2	-0031	F
1	0032	F
0	-0000	F

01:12
205

11	0002	F
10	0028	F
9	0005	F
8	0011	F
7	0015	F
6	0021	F
5	0009	F
4	0251	F
3	0064	F
2	0341	F
1	0682	F
0	0007	F

02:09
774

11	0000	F
10	0030	F
9	0010	F
8	-0013	F
7	0019	F
6	-0031	F
5	0031	F
4	-0031	F
3	0020	F
2	-0032	F
1	0031	F
0	0000	F

Sample 9, page 4

END

03:11
827.9

	11	0000	F
1444	10	-0032	F
1011	9	0021	F
1289	8	0028	F
1407	7	0031	F
1441	6	0032	F
1453	5	0033	F
1451	4	0031	F
1475	3	0033	F
1475	2	0032	F
1481	1	0032	F
	0	0000	F

02:51
819.4

	11	-0000	F
1440	10	0032	F
960	9	-0019	F
1264	8	-0027	F
1386	7	0030	F
1440	6	0032	F
1464	5	0031	F
1454	4	0031	F
1476	3	0032	F
1475	2	0032	F
1481	1	0031	F
	0	-0000	F

3:06

11	0000	F
10	0031	F
9	0021	F
8	-0028	F
7	0030	F
6	0032	F
5	0032	F
4	0032	F
3	0033	F
2	0033	F
1	0033	F
0	0000	F

02:41
813

	11	0000	F
1437	10	-0031	F
907	9	0019	F
1234	8	0026	F
1354	7	0029	F
1438	6	0031	F
1467	5	0032	F
1454	4	0032	F
1476	3	0031	F
1475	2	-0032	F
1481	1	0032	F
	0	-0000	F

03:01
824.4

	11	0000	F
1443	10	0031	F
991	9	0020	F
1280	8	0028	F
1400	7	-0031	F
1441	6	0032	F
1458	5	0032	F
1453	4	0032	F
1475	3	0032	F
1475	2	0033	F
1482	1	0032	F
	0	0000	F

Sample 10, Page 1

00:31
7

10	0117	
9	0117	
8	0161	
7	0092	
6	0125	
5	0111	
4		
3	0548	F
2	0705	F
1	1136	F
0	0077	F

3	0502	F
2	0631	F
1	1042	F
0	0076	F

00:52
46

10	0206	
9	0218	
8	0280	
7	0204	
6	0189	
5	0235	
4		
3	0560	F
2	0789	F
1	1297	F
0	0040	F

3	0547	F
2	0770	F
1	1281	F
0	0064	F

Time 00:20
M- 3

10	0090	
9	0099	
8	0131	
7	0085	
6	0088	
5	1088	
4	~~0447~~	F
3	0450	F
2	0550	F
1	0934	F
0	0076	F

00:41
17

10	0205	
9	0143	
8	0208	
7	0188	
6	0201	
5	0209	
4		
3	0598	F
2	0798	F
1	1241	F
0	0063	F

time=0

3	76
2	76
1	73
0	64?

Sample 10, Page 2

01:18
224

10	0246	
9	0344	
8	0467	
7	0216	
6	0210	
5	0544	
4	0390	
3	0827	
2	1172	
1	1480	
0	0000	F

01:50
577

10	770	
9	457	
8	684	
7	262	
6	799	
5	1299	
4	661	
3	984	
2	1400	
1	1480	
0	0004	F

01:08
142

10	0209	
9	0293	
8	0384	
7	0210	
6	0204	
5	0324	
4	0273	
3	0776	
2	1096	
1	1481	
0	0009	F

01:39
444

10	571	
9	423	
8	611	
7	234	
6	485	
5	1123	
4	582	
3	933	
2	1322	
1	1480	
0	0009	F

0	0002	F

Sample 10, Page 3

02:31
991

10	1373	
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8	743	
7	1204	
6	1388	
5	1458	
4	809	
3	1067	
2	1473	
1	1480	
0	0001	F

02:21
946

10	1293	
9	537	
8	733	
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6	1288	
5	1441	
4	807	
3	1062	
2	1477	
1	1480	
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02:10
856

10	1133	
9	514	
8	748	
7	786	
6	1159	
5	1430	
4	793	
3	1059	
2	1487	
1	1480	
0	0007	F

03:23
1007

10	1408	
9	882	
8	1219	
7	1353	
6	1415	
5	1458	
4	817	
3	1474	
2	1474	
1	1480	
0	-0000	F

03:02
1007

10	1388	
9	669	
8	1005	
7	1232	
6	1401	
5	1465	
4	809	
3	1388	
2	1475	
1	1480	
0	-0000	F

02:52
1007

10	1382	
9	572	
8	809	
7	1207	
6	1397	
5	1462	
4	804	
3	1207	
2	1474	
1	1480	
0	0000	F